

REMARKS

Support for the claims 25 through 58 is found in the specification at the locations indicated in the following chart.

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CLAIM	SUPPORT
Claim 25	page 7 and example 6 at pages 19 and 20
Claims 26 and 27	page 11, lines 18 and 20
Claims 28-31	page 7, lines 10 through 15
Claims 32 through 35	page 6, lines 25 through 30
Claims 36 and 37	page 7, line 10
Claim 38	Page 7 lines 24 through 30
Claim 39	page 10, lines 1 through 4
Claim 40	page 7, lines 7-18
Claim 41	page 8, lines 11-12
Claim 42	page 8, lines 25 through 26
Claim 43	page 5, lines 31 through 32
Claim 44	page 4, lines 1 through 31 and page 5, lines 1 through 32 and page 6, lines 5 through 15 and 25 through 30 and page 7, lines 15 through 23
Claim 45	page 11, lines 15 through 20
Claims 46 through 48	Same location as previously submitted claims 3 through 5
Claim 49	page 5, lines 23 through 26
Claim 50	page 5, lines 26-27 and 17-25
Claims 51 and 52	page 6, lines 25-30
Claim 53	page 15, lines 10-15
Claim 54	page 7, lines 26 through 30
Claim 55	page 10, lines 1 through 5
Claim 56	page 7, lines 17-18
Claim 57	page 8, lines 11-12
Claim 58	page 8, lines 25 through 26

The specification has been amended to delete inaccurate punctuation marks at the following pages: 4, 16, 17, and 19.

The Applicant's attorney hereby affirms the election for continued prosecution of claims or the subject matter of claim 1-13. Similar subject matter differing in scope is captured in newly presented claims 25 through 58.

Previously submitted claims 3, 8 and 12 were rejected under 35 U.S.C. 112 first paragraph for the use of the language glass transition temperature. Newly presented claim 46 utilizes the terminology “glass transition temperature” for the adhesive composition. It was noted in the rejection under 112 first paragraph for the previously submitted claims 3, 8 and 12 that the glass transition temperature is known in the art as directed to the property of thermoplastics and rubbers not to a composition having various components. The office action further noted that the glass transition temperature can be calculated by using the fox equation for organic polymers and copolymers. The instant dehydrating agent such as vinyl trimethoxysilane taught at page 17 line 17 is a monomer and monomers (and inorganic fillers) do not have a glass transition temperature. Thus applicant failed to describe adequately how to measure and obtain the glass transition temperature of the adhesive composition.

It is respectfully submitted that glass transition temperatures are known in the art as measurements for compositions such as food and adhesives and polymeric compositions. The enclosed copy of the Power Point presentation entitled “Drying of Sticky Materials - Relevance to Glass Transition Temperature (Tg)” by Dr. Vhesh Bhandari of the Food, Science and Technology School of Land and Food Sciences University of Queensland, Australia available at www.fst.uq.edu.au/staff/bbhandari/SprayDryingGroup/KMUTTThailand.ppt shows obtaining glass transition temperatures in the chart entitled “Physical Properties of Sugars and Stickiness Behavior For Organic Compounds That Are Sugars” and in the table entitled “Glass Transition Temperature of the Various Food Materials for Other Organics Including Citric Acid, Starch, Ice Cream and Honey”. Also, the technical paper entitled “Effects of Tg and CTE on Semiconductor Encapsulants” by Dr. Mark N. Konarski of Loctite Corporation, available at www.loctite.com is also enclosed herewith which indicates obtaining glass transition temperatures for epoxy encapsulating compositions for electric circuit boards. These epoxy compositions contain numerous ingredients. Also, a search was done of the U.S. Patent and Trademark Office database for

specifications having the word string "glass transition" and claiming the word "adhesive" and the word string "glass transition" and 985 issued patents were listed. Another search was done on the same database for patents having specifications with the word string "glass transition" and claims having the word 5 "adhesive" and the word string "glass transition" and also specifications having the words either "measure the glass" or "measure of the glass" which uncovered one patent, U.S. Patent, 4,814,373. This latter search was altered by modifying the last section of the search for patents having specifications using the word string "measurement of the glass" and uncovered the following thirteen patents.

10 PAT. NO. Title

- 1 6,605,347 Pressure-sensitive adhesive composition and pressure-sensitive adhesive sheet thereof
- 2 6,558,791 Heat-resistant adhesive sheet
- 3 6,498,203 Rubber-erasable aqueous ink for writing material composition and writing materials using the same
- 15 4 6,372,080 Process for fabricating a crack resistant resin encapsulated semiconductor chip package
- 5 6,248,613 Process for fabricating a crack resistant resin encapsulated semiconductor chip package
- 6 6,228,500 Adhesive composition and precursor thereof
- 7 6,057,384 Latex polymer blends for improving the permanence of ink-jet inks
- 8 6,046,072 Process for fabricating a crack resistant resin encapsulated semiconductor chip package
- 20 9 5,990,202 Dual encapsulation technique for preparing ink-jets inks
- 10 5,461,087 Adhesive and double-sided adhesive tape
- 11 5,089,346 Heat resistant adhesive composition and bonding method using the same
- 12 4,624,893 Pressure-sensitive adhesive tapes or sheets
- 13 4,500,575 Hot-melt adhesive of a copolyester of a dibasic acid with a polyhydric alcohol

25 Therefore, it is respectfully submitted that those skilled in the art are well aware of ways of determining glass transitions of compositions ranging from those full of ingredients such as ice cream to those of epoxy compositions for encapsulating integrated circuits or circuit boards.

30 Previously submitted claims 1 through 13 were rejected under 35 U.S.C. 112 second paragraph as being indefinite for the recited terminology "the clarity of claim 1." and also for the word "about" in claims 3, 6-9 and 12 as indefinite in regards to the terminology "less than". In claims in newly presented claims 25 through 58 using similar terminology to that of previously claims 3, 6-9 and 12 35 have not used the term "about" in relation to the words "less than". In regards to the use of the word "the clarity" similar words are used in claim 44 as "interfere with the production of a clear adhesive". It is respectfully submitted that this latter terminology has antecedent basis for claim 44.

Previously submitted claims 10-13 were rejected under U.S.C. 102(b) as anticipated by or in the alternative under 35 U.S.C. 103(a) as obvious over the product brochure entitled MS Polymer Silyl of Kaneka Corporation. It is respectfully submitted that the two independent claims, claim 25 and claim 44 and claims dependent therefrom are novelty and unobviousness from the teaching of this reference. Claim 25 has a filler which includes fumed silica with a surface area of less than 200 meters squared per gram in addition to the polymer or copolymer with reactive silicone end groups and the dehydrating agent. Always having the presence of such a filler which is fume silica is not taught or suggested by the Kaneka reference. Also for claim 44, there is no teaching or suggestion in the Kaneka reference to have the filler of fume silica with a certain surface area, or a clear filler with a certain surface area and a viscosity range for the adhesive.

Previously, claims 1-13 were rejected under 35 U.S.A. 103(a) as obvious over the product brochure "MS Polymer Ssilyl" of Kaneka Corporation in view of the Smith Jr. et. al. U.S. Patent 4,308,372, Staiger et al (U.S. Patent 5,304,621) and/or Imai et al (U.S. Patent 4,760,123). It is noted in the office action that fillers are generally taught at the bottom of page 1 of the product brochure and the other patents used in the rejection use the fumed amorphous silica in moisture curable compositions having copolymer with reactive silicone and groups. For example, it is noted in the office action that Staiger et al referenced taught fumed silica, HDK, at column 18 line 7 which was also taught in the specification of the captioned application at page 6 line 22. A conclusion of the office action was it would be obvious to one skilled in the art to take the fumed silica of Smith, Staiger or Maimai references and use it as a filler for the teaching of the product brochure with the particular polymer or copolymer in a moisture curable composition.

It is respectfully submitted that none of the aforesaid references teach the polymer or copolymer with the reactive silicon groups with the particular type of filler and the dehydrating agent as an adhesive composition.

It is respectfully submitted that the newly presented claims are in condition for allowance and reconsideration and allowance of them is earnestly solicited.

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Respectfully submitted,

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DRYING OF STICKY MATERIALS- RELEVANCE TO GLASS TRANSITION TEMPERATURE (T_g)

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Stickiness issues:

- Stickiness on the drier wall (spray drying)**
- Wet and plastic appearance**
- Agglomeration and clumping in packing container**
- Operational problems**

Products exhibiting stickiness

Products with high amount of sugars or organic acids

- Fruit juices/pieces/purees/leathers**
- Honey**
- Molasses**
- Whey (acid or sweet)**
- High DE maltodextrins (DE>30)**
- Pure sugars- lactose, glucose, sucrose, fructose**
- High acid foods**

High fat foods

Major factors causing stickiness

- High hygroscopicity**
- High solubility**
- Low melting point temperature**
- Low glass transition temperature
(related to thermoplasticity)**

Glass Transition Approach

- Recent approach to describe stickiness**
- Applied to spray drying**

Physical properties of sugars and stickiness behaviour

Sugars	Hygroscopicity (relative)	Melting point (°C)	Approx solubility in H ₂ O 60°C (% w/w)	Tg (°C)	Stickiness (relative)
Lactose	+	223	35	101	+
Maltose	++	165	52	87	++
Sucrose	+++	186	71	62	+++
Glucose	+++++	146	72	31	+++++
Fructose	++++++	105	89	5	++++++

What is a glass transition?

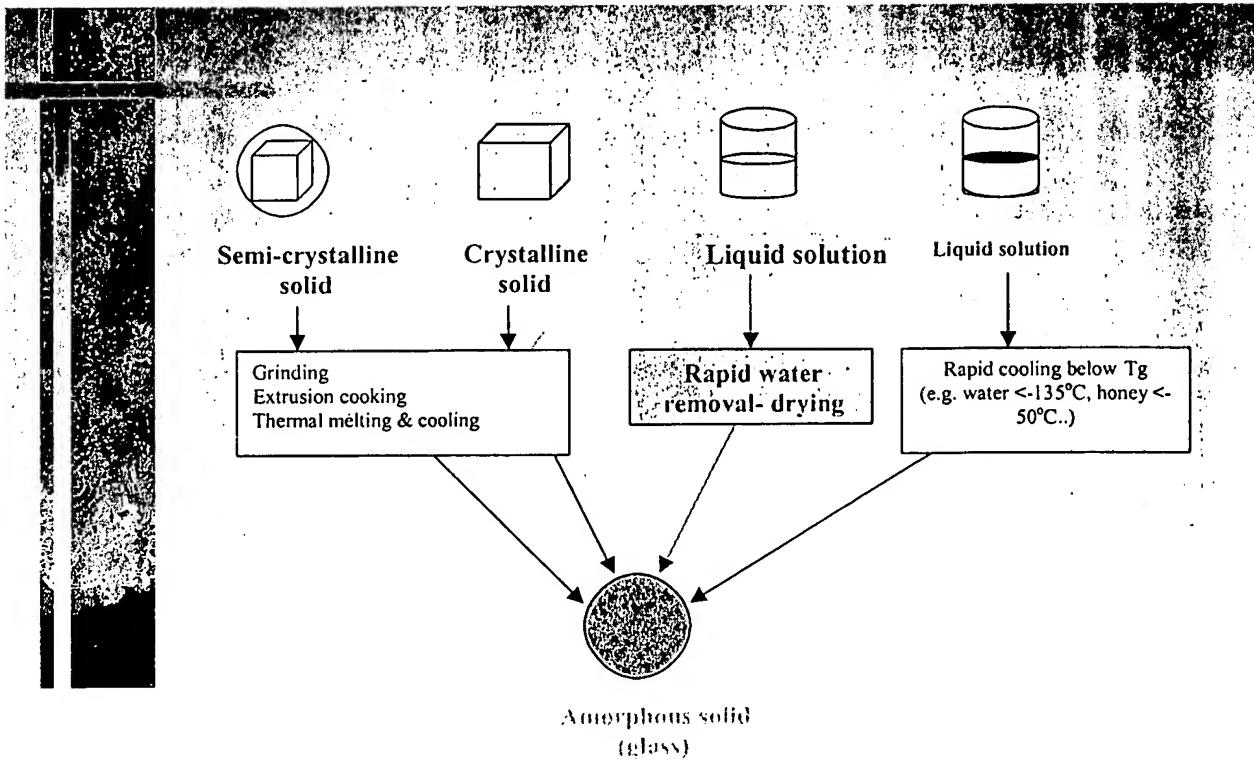
Physical states of dried or drying solid materials:

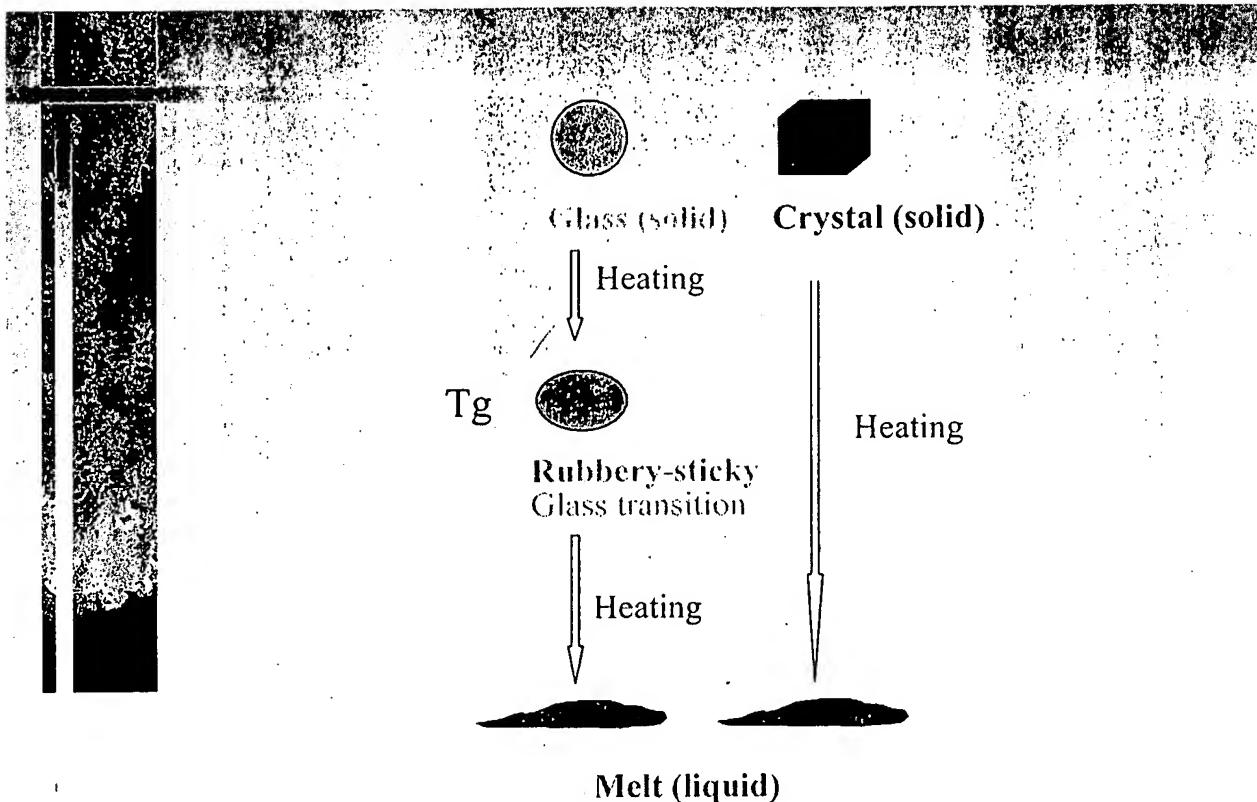
— Amorphous

- non-aligned molecular structure
- very hygroscopic
- go through glass transition
- predominant in dried food

— Crystalline

- aligned molecular structure
- non hygroscopic
- no glass transition





General concepts

- If the product temperature is above its glass transition temperature (Tg)- it will exhibit stickiness
- Shorter chain molecules- low glass transition temperature (Tg of monosaccharides < Tg of disaccharides)
- Water depresses the Tg significantly (Tg of amorphous solid water is -135°C)
- For a complex food system, the Tg is a function of weight fraction of each component and their Tgs'- but the relationship is not linear

Glass transition temperature of various food materials

Food materials	T _g (°C) ^{abc}
Fructose	5
Glucose	31
Galactose	32
Sucrose	62
Maltose	87
Lactose	101
Citric acid	6
Tartaric acid	18
Malic acid	-21
Lactic acid	-60
Maltodextrins	
DE ^d 36 (MW=550)	100
DE 25 (MW=720)	121
DE 20 (MW=900)	141
DE 10 (MW=1800)	160
DE 5 (MW=3600)	188
Starch	243 ^e
Ice-cream	-34.3
Honey ^g	-42 to -51

Glass transition related problems in various drying processes

- **Spray drying:** sticking on the drier wall, duct and cyclone, poor recovery of powder, agglomeration in the collection bag or container
- **Freeze drying:** “Collapse” of structure while drying
- **Conventional hot air solid drying:** poor fluidisation, stick on the drying racks/shelves, soft product while drying but solid after cooling
- **Storage:** Clumping, agglomeration, caking, crystallisation

Some solutions

- **Drying below the glass transition temperature (often not feasible)**
- **Choosing mild drying temperature conditions**
- **Increasing the T_g of the food by adding high molecular weight materials (such as maltodextrins)- a predictive approach according to the composition**
- **Immediate cooling of the product below its T_g**
- **Appropriate drier design to suit the sticky product**

Conclusion

- At the glass transition temperature the amorphous food is converted to rubbery state (from its solid glassy state)
- If the temperature of the product is above its glass transition temperature it exhibits stickiness
- The stickiness can be minimised by lowering drying temperature and increasing the T_g by adding high molecular weight additive
- An optimisation procedure is needed to control the T_g of the product and to select correct drying conditions



Electronics

TECHNICAL PAPER

Effects of Tg and CTE on Semiconductor Encapsulants

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www.Loctite.com

Abstract

As the role of direct-chip-attachment increases in the electronics industry, the reliability and performance of COB packaging materials becomes an increasing concern. Although many factors influence component reliability, the biggest determinants of performance are often the glass transition temperature (Tg) and the coefficient of thermal expansion (CTE) of the encapsulant or underfill. This paper discusses exactly what these properties are, how they are measured, and why they are important to device-reliability. Both materials and processes can impact these thermal properties. The nature of acrylate UV-cured materials versus thermally cured epoxies is examined and the significance of different cure parameters is discussed.

Introduction

The glass transition temperature and coefficient of thermal expansion of chip encapsulants have direct effects on semiconductor package performance and reliability.¹ Though they both are usually quoted and accepted as single numerical values, different methods of measurement and sample preparation will provide varying data for the same material. Even within the same sample, the glass transition occurs within a range of temperatures and not as a single point. Factors such as intrachain stiffness, polar forces, and comonomer compatibility can affect the size of the glass transition region.² Thermal expansion coefficients are affected by polymer chain stiffness, packing, cure shrinkage and filler interactions.³

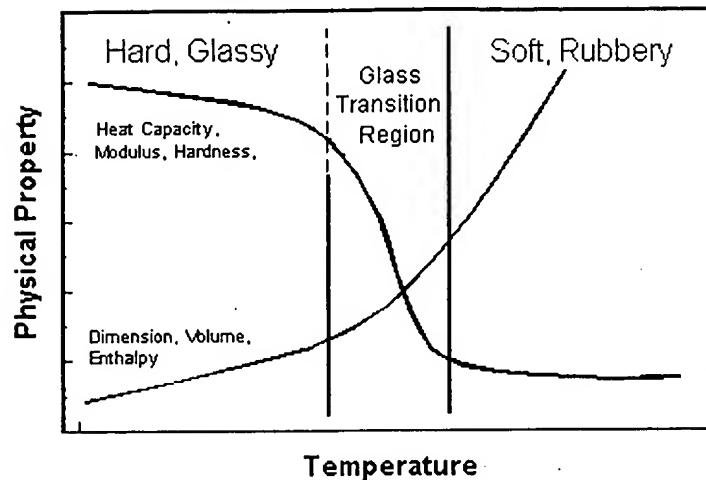
The purpose of this paper is to provide a summary of the common practices and techniques involved with Tg and CTE. Methods of measurement are explained and compared. Differences in sample preparation are shown to significantly affect results and suggest concerns about the performance of microelectronic packages. A better understanding of the factors affecting these material properties will allow process and design engineers to gain optimum performance from packaging encapsulants and become cognizant of the potential reliability changes that can occur from changes in board-level processing.

Instrumental Measurement

The glass transition temperature ("Tg") is the point where a substance changes from a hard-glassy material, to a soft-rubbery one. In monomer or thermoplastic polymers, the transition is from a solid or glass to a flowable liquid. For crosslinked thermosetting polymers, the transition is to a soft-rubbery composition and tends to occur across a thermal band rather than at a distinct point of temperature.

At the glass transition temperature, several easily measurable properties such as volume, dimension, enthalpy, strength and modulus also undergo transitions, and are often used to determine Tg's. Figure 1 schematically portrays the shifts in the glass transition region.

FIGURE 1
PROPERTY CHANGE



The property shifts at the glass transition temperature of encapsulants can cause severe stress buildup in packaged components leading to premature device failures¹. Because of this, the T_g of the encapsulant usually places a limit on the use temperature and therefore becomes an important consideration when specifying for a particular application. Chemical composition and extent of cure are the major influences on T_g, however, the method of measurement can vary widely and will affect the data. The most commonly used analytical methods are compared in the following table.

Analytical Method Comparison

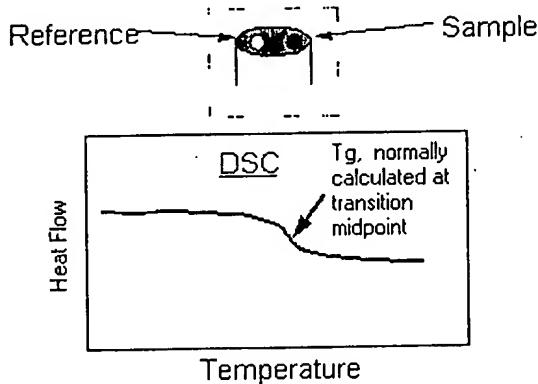
	Typical Run Time	Sample prep	Repeatability	Dependability	Comments
<u>DSC</u>	20 minutes	easy	good	marginal	Many materials do not exhibit clear transitions.
<u>TMA</u>	40 minutes	medium	fair	good	Very dependant on sample preparation.
<u>DMA</u>	120 minutes	difficult	excellent	excellent	T _g can be defined several different ways.

The coefficient of thermal expansion is a measure of the fractional change in dimension (usually thickness) per degree rise in temperature. For microelectronics encapsulants, it is often quoted in "ppm/°C" (value x 10-6/°C). Chemical composition, filler loading and cure cycles all affect the value. For typical materials that have non-linear expansion, the

specified temperature range will also have an effect on the data, with measurements out closer to the Tg yielding higher values than those quoted across lower temperature bands.

Differential scanning calorimetry (DSC) is the quickest and simplest test for Tg. The method requires extremely small samples (typically 5-20 mg) that require no special preparation, and material from components or processed boards can be utilized. The method consists of heating the sample in a closely calibrated thermocouple where the temperature of the sample is compared to the temperature of a blank reference point within the same cell. Thermodynamic transitions such as melting points and reaction exotherms are easily measured, and the change in heat capacity at the Tg is seen as a shift in the baseline for cured encapsulants as shown in Figure 2.

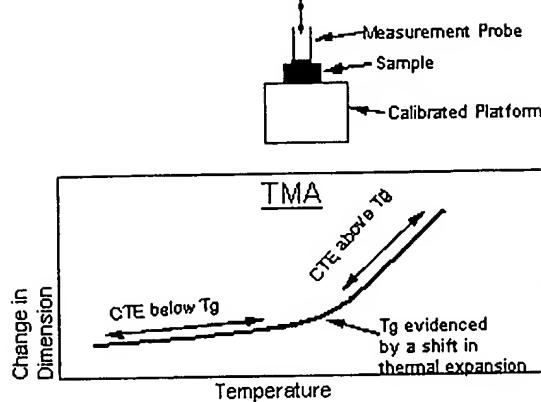
FIGURE 2
Differential Scanning Calorimetry



Unfortunately, this fast and convenient method is not universally applicable to all materials. High filler loadings, high crosslink densities, and other thermo-molecular processes can mask the shift due to the Tg and make the transition difficult or impossible to identify.

Thermo mechanical analysis (TMA) is the test used to determine thermal expansion coefficients. Since there is a shift to a higher thermal expansion coefficient above the Tg due to changes in molecular free volume⁴, the method can also be used to measure the glass transition temperature as shown in Figure 3.

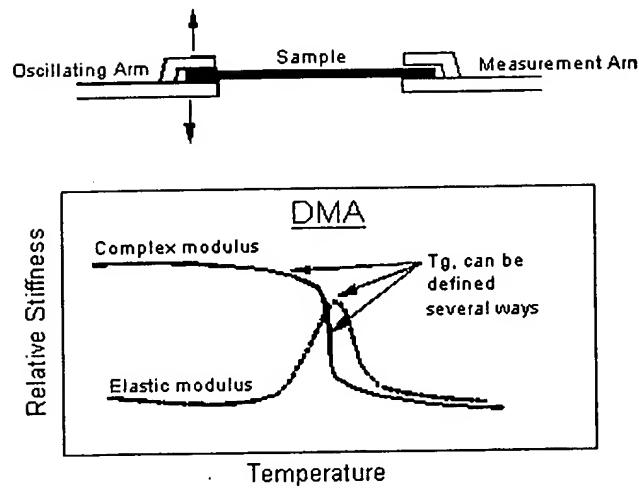
FIGURE 3
Thermo Mechanical Analysis



The technique simply consists of heating the sample upon an expansion-calibrated platform and measuring the dimensional change of the sample with an instrumented probe. The method will also easily follow cure-stress relaxations in and around the glass transition region which sometimes leads to ambiguity in assignment of a specific Tg and can yield a different value for the same specimen if measured at a different point (for instance, the cure stress may be significantly different when measured near the edge of a sample versus its center).

Dynamic mechanical analysis (DMA) consists of oscillating flexure energy applied to a rectangular bar of the cured encapsulant. The stress that is transferred through the specimen is measured as a function of temperature. Components of material stiffness are separated into a complex modulus and a rubbery modulus. The technique is highly accurate although the Tg can be defined in different ways which will have different values as illustrated in Figure 4.

FIGURE 4
Dynamic Mechanical Analysis



Large samples that must be accurately machined, coupled with longer setups make this test relatively expensive to run.

Each of the methods will produce different data for the same material as shown in the following table.

Instrument Effect on Glass Transition Temperature

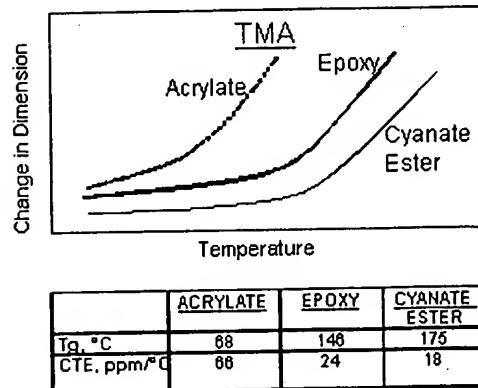
INSTRUMENT	GLASS TRANSITION TEMPERATURE
DSC	142°C
TMA	130°C
DMA	137°C (G") 146° (Tan delta)

A single specimen of a developmental epoxy encapsulant was cast and cured for 2 hours at 145°C after gelling at 100°C for one hour. It was then machined into specimens for DSC, TMA and DMA analysis. The results show Tg's ranging from 130°C for the TMA, to 146°C for the DMA measurement.

Discussion

The most obvious effect on the glass transition temperature and thermal expansion coefficient comes from the chemistry involved in the encapsulant. Figure 5 depicts a comparison of experimental epoxy, acrylate, and cyanate ester encapsulants.

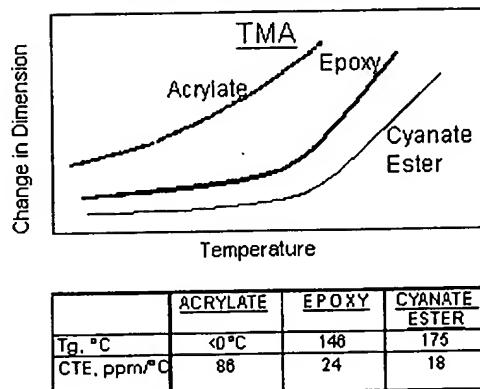
FIGURE 5
Chemistry Effects



All three were loaded with 75% of a proprietary low-expansion filler combination and thoroughly cured. The acrylate is a UV-curing system which incorporates a high flexibility/low modulus polymer backbone as evidenced by its low Tg. This sort of molecular architecture results in a relatively high CTE, even when the encapsulant is loaded with low-expansion filler. The epoxy is typical of current high reliability materials offering a reasonable Tg/CTE combination while the cyanate ester provides further improvements.

The following table shows the thermal expansions coefficients for the different materials encountered at the board level.

FIGURE 5
Chemistry Effects



Component stress from expansion mismatches is dispersed through the encapsulant when it closely matches the CTE of the board and associated metals. Stress is amplified and compounded when the encapsulant's CTE is high. Lowering the thermal expansion of chip on board encapsulants in order to more closely match expansions of the board, wafer, and solder junctions has been shown to have a direct effect in increasing package reliability. Since all useable encapsulant polymers have expansion coefficients several times larger than the materials to be protected, low expansion fillers are added. The filler loading level is normally as high as possible while retaining the necessary processability. The following table depicts the effect of different filler loading levels within the same material.

Effect of Filler Loading in an Epoxy Matrix

FILLER LOADING	TMA-Tg	CTE (25-100°C)
60 %	129°C	34.5 PPM/°C
65 %	127°C	28.8 PPM/°C
70 %	132°C	24.1 PPM/°C
75 %	130°C	20.0 PPM/°C
80 %	Unprocessable	..

The matrix consists of a standard epoxy anhydride which is loaded with a proprietary mixture of low expansion fillers. All samples were cured for 2 hours at 145°C after gelling for 1 hour at 100°C. CTE's decrease rapidly and the viscosity increases while the effect on Tg is negligible.

Cure temperature affects the extent of cure, the rate of gellation and the cure stress which is a function of the gellation temperature. As a result, the cure temperature influences both the coefficient of thermal expansion and the Tg of the encapsulant. As cure temperature increases, crosslink density and degree of cure increase. Since the glass transition is a function of both, the Tg undergoes a corresponding rise until a point where nearly all polymer reaction sites have been used. At that point, the glass transition temperature plateaus with further temperature increase unless thermo-degradation begins. The following table illustrates the point, where increasing the cure temperature from 130°C to 175°C increases the Tg of the encapsulant from 117°C up to 146°C. At the same time, the CTE increases from 22.9 ppm/°C to 24.3 ppm/°C.

Cure Temperature Effects

CURE TEMP	TMA-Tg	CTE (25-100°C)
130°C / 2 HOURS	117°C	22.9 ppm/°C
145°C / 2 HOURS	127°C	23.8 ppm/°C
160°C / 2 HOURS	137°C	26.3 ppm/°C
175°C / 2 HOURS	148°C	24.3 ppm/°C
PREGEL + 130°C	113°C	21.1 ppm/°C
PREGEL + 145°C	130°C	20.0 ppm/°C
PREGEL + 160°C	138°C	19.8 ppm/°C
PREGEL + 175°C	140°C	19.4 ppm/°C

The increase in coefficient of thermal expansion is due to higher levels of cure stress being built into the samples as the temperature increases. The cure stress is a function of gellation temperature because the faster that the material gels, the more likely it is for polymer chains to become locked into stressed configurations and the more that shrinkage will occur as the sample cools after cure. Since it is ultimately desirable to have both a high Tg and a low CTE, encapsulants are often gelled at temperatures significantly lower than the cure temperature. Once the encapsulant has gelled, more time at higher temperatures actually decreases stress through an annealing process and results in significantly lower CTE's.

The amount of time at temperature also has an effect. The following table shows the results of cure time experiments carried out on a developmental anhydride-cured epoxy encapsulant.

Cure Temperature Effects

CURE TEMP	TMA-Tg	CTE (25-100°C)
130°C / 2 HOURS	117°C	22.9 ppm/°C
146°C / 2 HOURS	127°C	23.8 ppm/°C
160°C / 2 HOURS	137°C	26.3 ppm/°C
175°C / 2 HOURS	146°C	24.3 ppm/°C
PREGEL + 130°C	113°C	21.1 ppm/°C
PREGEL + 146°C	130°C	20.0 ppm/°C
PREGEL + 160°C	138°C	19.8 ppm/°C
PREGEL + 175°C	140°C	19.4 ppm/°C

Between 0.5 and 2.0 hours at 160°C, Tg and CTE both increase with the increasing extent of cure. Considerable cure stress is built up in the process as discussed earlier. Once the material is fully cured, additional time at temperature allows a slow relaxation of chains trapped in stressed configurations resulting in significant lowering of the CTE without further increase in Tg. Although this type of cure is unrealistic from a processing standpoint, it does illustrate the type of performance that can be ultimately achieved.

Summary

The glass transition temperature and coefficient of thermal expansion for COB encapsulants have been shown to not exist as single numbers, but as ranges of values that are very dependent on measurement techniques and processing methodology. Designing from data developed under different conditions than those seen at the board level will very likely be in error and caution is urged. Cure parameters have a significant impact on the data, and should be noted for any reference to a material's CTE or Tg. Property comparisons must be made on materials that have been processed and tested in similar manners to be meaningful.

References

1. Voloshin, A.S. and Tsao, P.H., "Analysis of the Stresses in the Chip's Coating" Proceedings of the 43rd Electronic Component and Technology Conference, (1993).
2. Roller, Mark B., "The Glass Transition: What's the Point?", J. Coating Technology, (1982), 54(691) pp 33-40.
3. Fahmy, A.A. and Raga, A.N., "Thermal-Expansion Behavior of Two-Phase Solids", J. Appl. Phys., (1970), 41 pp 5108-5111.
4. Goldstein, M. "Some thermodynamic Aspects of the Glass Transition: Free Volume, Entropy and Enthalpy Theories", J. Chem. Phys., (1963), 39 pp 3369-3374.

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